

Evaluation of Atomic Oxygen Resistant Protective Coatings for Fiberglass-Epoxy Composites in LEO

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EVALUATION OF ATOMIC OXYGEN RESISTANT PROTECTIVE COATINGS
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Abstract

Fiberglass-epoxy composite masts are the prime structural members for the Space Station Freedom solar array. At the altitude where Space Station Freedom will operate, atomic oxygen atoms are the most predominant species. Atomic oxygen is highly reactive and has been shown to oxidize organic and some metallic materials. Tests with random and directed atomic oxygen exposure have shown that the epoxy is removed from the composite exposing brittle glass fibers which could be easily removed from the surface where they could contaminate Space Station Freedom Systems. Protection or fiber containment systems; inorganic based paints, aluminum braid, and a metal coating; were evaluated for resistance to atomic oxygen, vacuum ultraviolet

radiation, thermal cycling, and mechanical flexing. All appeared to protect well against atomic oxygen and provide fiber containment except for the single aluminum braid covering. UV radiation resistance was acceptable and in general, thermal cycling and flexure had little to no effect on the mass loss rate for most coatings.

1. INTRODUCTION

Fiberglass-epoxy composites have many potential space uses such as a mast material for Space Station Freedom. Space Station Freedom will utilize photovoltaics for the primary power generation system in the first phase of operation [1]. The photovoltaic cells are mounted to a flexible polyimide Kapton HN[®] blanket which provides support for the cells as

well as the interconnecting circuitry. A fiberglass-epoxy mast is used to extend and retract the solar array blanket and provide structural support. There are two main fiberglass-epoxy components to the mast: the battens and the longerons. There are three longerons per mast. They are continuous and extend the full length of the array (approximately 30.5 m (100 ft)). As the mast is retracted, the longerons coil into a canister for storage where they are under conditions of significant flexural stress. Battens are the cross braces which provide internal support to the mast and are under no stress when stored. A more complete description of the solar array assembly system is given in reference 2.

When extended, the mast components will be exposed to the low Earth orbital environment. The main constituent of this environment between 180 and 650 km (97 and 351 nmi) is atomic oxygen [3]. Impact of surfaces with energetic (4.2 to 4.6 eV) [4] atomic oxygen causes surfaces to oxidize which could lead to contamination of surrounding surfaces or structural failure. Many materials including epoxy are known to be susceptible to reaction with atomic oxygen [4]. In addition to atomic oxygen, durability to other environmental hazards such as UV radiation [5], and thermal cycling [5] need to be assessed for spacecraft materials.

This paper will discuss the effect of atomic oxygen on fiberglass-epoxy composites and several candidate protective coatings and coverings. The consequences

of thermal cycling, flexing, and UV radiation will also be discussed on both protected and unprotected fiberglass-epoxy surfaces. Testing concentrated on longerons since they undergo the largest amount of mechanical stress. Any technique which can provide protection for the longerons should also provide adequate protection for the battens.

2. EXPERIMENTAL PROCEDURE

2.1 Longerons and Protection Techniques

Longerons were supplied by AEC Able Engineering for testing. The longerons had rectangular cross-sections .64 by .74 cm (.25 by .29 in.) with rounded edges. Samples for atomic oxygen plasma exposure, flexure testing, and thermal cycling were 12.7 cm (5 in.) in length while samples for UV exposure and directed atomic oxygen beam exposure were 2.54 cm (1 in.) in length. Longerons contained 19 to 22 percent amine cured epoxy resin by weight; the remainder being S-2 glass fibers.

The protection systems evaluated for the fiberglass-epoxy are aluminum braid and double aluminum braid coverings (AEC Able Engineering); In-Sn eutectic coating of primer/electroless nickel/immersion coated gold/indium-tin eutectic (applied by Composite Optics Inc.); CV-1144, a silicone based paint (manufactured by McGhan Nusil and brush applied at NASA LeRC); and S13G/LO-1, a thermal control paint composed of potassium silicate treated zinc oxide pigment in a silicone elastomer vehicle

(manufactured by IIT Research Institute and applied by paint spray gun at NASA LeRC on top of a brush applied organosilane ester primer). The appearance of the samples prior to exposure is illustrated in Figure 1.

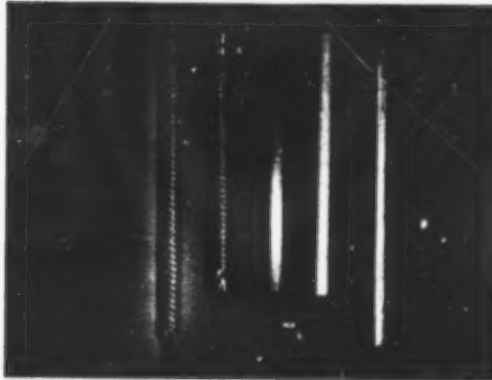


Figure 1. Protected Mast Samples (left to right): Single Al Braid, Double Al Braid, CV-1144, S13G/LO-1, and In-Sn Eutectic Covered

2.2 Dehydration

Samples were dehydrated in a vacuum chamber at a pressure between 2.7 and 9.3 Pa (20 mtorr and 70 mtorr) prior to mass measurement in order to eliminate errors due to absorbed water from the atmosphere and outgassing. The amount of time necessary for the sample to reach equilibrium ranged from approximately 5 days for unprotected fiberglass-epoxy to approximately 25 days for CV-1144 and S13G/LO-1 coated fiberglass-epoxy longerons. A detailed description of the dehydration exposure duration determination is given in reference 6.

2.3 Atomic Oxygen Exposure

Two types of atomic oxygen exposure systems were used for testing. An RF (13.56

MHz) air plasma asher (SPI Plasma Prep II) was used for durability testing of the longerons. Samples were exposed directly in the plasma at pressures ranging from 2.7 to 16 Pa (20 mtorr to 120 mtorr). The plasma contained atomic oxygen and nitrogen in various ionization and energy states. Although the various states of nitrogen are abundant, the reaction of materials with nitrogen appears to be negligible in relation to the reaction with the atomic oxygen states present [7]. Typical fluxes of atomic oxygen in the plasma ranged from 5×10^{14} to 2×10^{15} atoms/(cm²-sec). These values are based on the epoxy loss rate from the longerons assuming the same erosion yield for epoxy (1.7×10^{-24} cm³/atom) as measured in space on Shuttle flight STS-8 [8].

Although the flux is accelerated to what is experienced in low Earth orbit (LEO) and the plasma energy (a few 10ths of an eV) is lower, ashers can provide a good qualitative indication of material survivability in LEO. To date, materials that have survived in an asher have also survived in LEO. The asher was used to determine initial coating durability and to evaluate durability after thermal cycling and flexing. These tests were performed sequentially in order to better simulate the actual longeron environment. (Figure 2)

The arrival of atomic oxygen on the longerons in a plasma asher is from many angles while the atomic oxygen in space is nearly unidirectional. In order to determine what difference

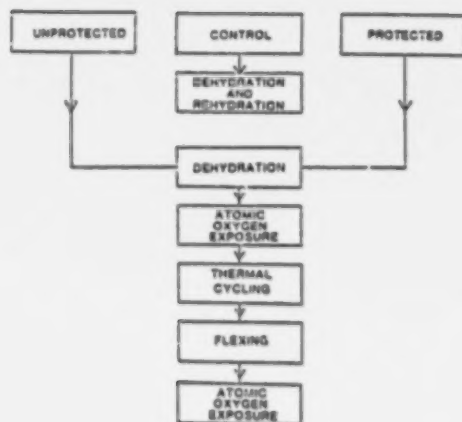


Figure 2. Longeron Test Sequence

directionality plays in the oxidation of fiberglass-epoxy, an atomic oxygen directed beam was used to expose smaller unprotected longeron samples for mass loss determination followed by scanning electron microscopy. The directed beam is produced by an end Hall gridless ion source (Commonwealth Scientific Inc.). A mixture of O^+ and O_2^+ is present in the beam which impinges on the samples at approximately 57 eV for an anode energy of 90 eV [9]. Pressure during operation was approximately $.013 \text{ Pa}$ ($1 \times 10^{-4} \text{ torr}$) with a base pressure of approximately $1.3 \times 10^{-4} \text{ Pa}$ ($1 \times 10^{-6} \text{ torr}$). Samples were exposed at an equivalent atomic oxygen fluence of approximately $9.4 \times 10^{20} \text{ atoms/cm}^2$ based on the erosion yield of epoxy in LEO.

2.4 Thermal Cycling

Thermal cycling was performed mainly to determine if thermal expansion mismatch between the protective surface and the longeron would cause cracks or openings to occur that would allow the

underlying surface to be exposed to atomic oxygen. Thermal cycling was performed at atmospheric pressure in a gaseous nitrogen environment by cycling the sample between a chamber heated by electric coils and a chamber filled with cold nitrogen gas. Transfer of the sample between chambers was controlled by a thermocouple embedded in the center of one longeron used only for temperature control. Up to three longerons could be exposed at one time on our sample tray. Each longeron experienced 413 thermal cycles between a core temperature of $+80$ and -80°C . The average time spent in one chamber was approximately 4 min.

2.5 Flexure Testing

Flexure testing was performed in order to simulate the extension and retraction of the mast from the mast canister. During storage, the longerons are coiled into a canister whose typical diameter is approximately 66 cm (26 in.). Longer on samples, both protected and unprotected, were flexed and then straightened with the aid of a clamping fixture to the radius of curvature they would experience in the canister for a total of 100 cycles. This corresponds to a surface strain per cycle of 0 to approximately 1 percent. The mast extension and retraction may occur only 10 to 20 times during the life of Space Station Freedom, so this is a more severe test of the mast flexural durability.

2.6 Ultraviolet Radiation Exposure

Longerons were exposed to vacuum ultraviolet (VUV) radiation followed by exposure to atomic oxygen in the asher to determine if VUV radiation would affect the protective coating such that cracks or defects would be formed in the coating and the underlying surface would be degraded by atomic oxygen. Samples were exposed on one side. VUV radiation was provided by a deuterium lamp in a water cooled chamber inside a vacuum belljar. Pressure during operation was 5×10^{-5} Pa (4×10^{-7} torr) to 2.7×10^{-4} Pa (2×10^{-6} torr). The sample exposure level was 5 equivalent suns for approximately 1000 equivalent sun hours (ESH). Sample temperature during exposure was between approximately 21 and 32 °C (70 and 90 °F). By using a deuterium lamp in vacuum for UV exposure, an accelerated UV exposure is achieved in the 100-200 nm (3.9×10^{-6} to 7.9×10^{-6} inches) wavelength range with a less than real time exposure closer to the visible region of the spectrum. Since the wavelength region that is accelerated contains the most damaging radiation, the test is more severe and UV damage should be more evident.

3. RESULTS AND DISCUSSION

3.1 Durability Testing of Unprotected Fiberglass-Epoxy

Unprotected fiberglass-epoxy longerons were exposed to atomic oxygen in a plasma asher for 1747 hr which corresponds to an equivalent atomic oxygen fluence of 8.8×10^{21} atoms/cm² based on the erosion yield of epoxy in LEO. The mass loss per unit

area for two longerons exposed at different spatial locations in the plasma was recorded as a function of time along with the mass loss per area of a sample of polyimide Kapton[®] included for comparison (Figure 3).

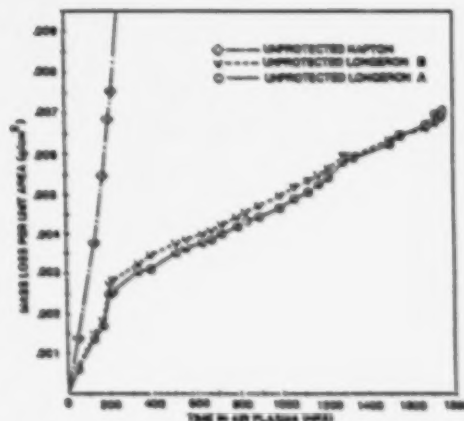


Figure 3. Mass Lost From Unprotected Fiberglass-Epoxy as a Function of Plasma Exposure Time

From previous asher tests and data obtained in space, it is known that glass and other metal oxides are very resistant to attack by atomic oxygen [8]. Therefore, the mass loss observed is most likely from the epoxy in the composite and not the S-2 glass fibers. The drastic change in slope that occurs at approximately 200 hr is consistent with the change in mass loss rate which would occur if the epoxy on the surface had been removed leaving a mix of epoxy and glass fibers on the surface in a proportion equivalent to the fill ratio. The most alarming thing about this data is that the mass loss rate does not eventually decrease as would be expected if the fiberglass on the surface shielded the

deep, visually hidden, underlying epoxy. This appears to indicate that the fiberglass-epoxy would not become self protecting once the glass fibers were exposed. For a 15 year exposure in a constant density environment for Space Station Freedom, the average fluence that the mast surfaces are likely to be exposed to would be approximately half of the front and back solar array exposure or 2.1×10^{21} atoms/cm² [10]. This exposure would correspond to approximately 4220 hours in the asher based on an equivalent asher fluence. Assuming that the oxidation depth is small in comparison to the longeron diameter, and the rate of epoxy loss remains constant beyond 1747 hours in the asher, the depth of oxidation can be approximated by calculating the loss from a flat fiberglass-epoxy surface with a surface area percentage of 31% epoxy after approximately 200 hours of asher exposure. In this manner, it was determined that the outer layer of epoxy covering the fibers for these particular samples was approximately .0016 cm (6.3×10^{-4} in.). The depth of oxidation below the outer epoxy layer was approximately .0285 cm (.011 in.). Since the glass fibers are approximately 10 microns (3.9×10^{-4} in.) in diameter, this depth would correspond to approximately 29 fiber diameters. In terms of overall depth, this would correspond to only 5% of the longeron diameter with an overall longeron mass loss of 2% over the life of Space Station Freedom. These numbers are not very large and would not cause great concern. However, the glass fibers which are exposed

could break off and contaminate Space Station Freedom systems, or could cause difficulty in mast retraction if the loose fibers created friction in the track of the extension and retraction mechanism. Figure 4 shows an unexposed fiberglass-epoxy longeron and one that was exposed to atomic oxygen in the plasma asher for an equivalent fluence of 8.8×10^{21} atoms/cm² which is approximately 41% of the Space Station Freedom life. Individual fibers can be seen that have lifted off of the surface.

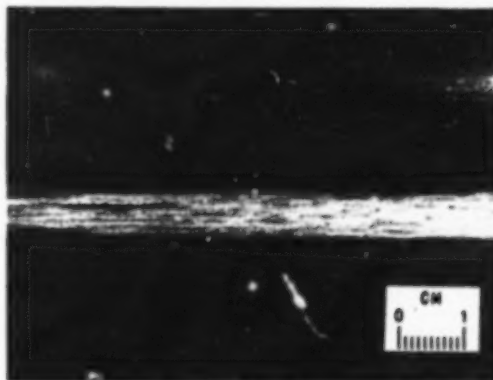
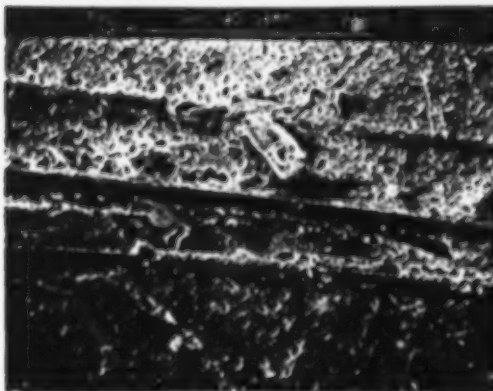


Figure 4. Unexposed (Top) and Plasma Exposed (Bottom) Fiberglass-Epoxy

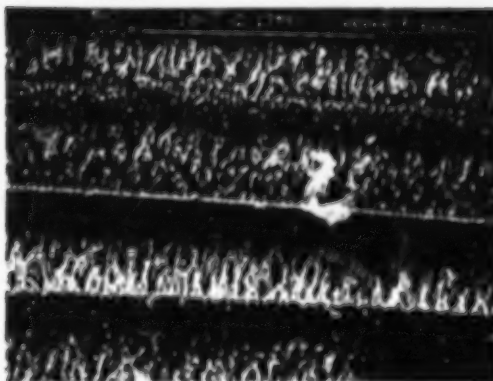
The actual depth of oxidation and amount of fiber exposure may not be as severe in LEO since the plasma atomic oxygen exposure is omnidirectional while the actual exposure would be better simulated by a sweeping directed beam. Therefore, a directed beam of atomic oxygen ions was used to bombard the samples to determine if there are differences between directed and random exposure. Scanning electron photomicrographs in Figure 5 show fiberglass-epoxy exposed to the air plasma at an equivalent fluence of 5×10^{20} atoms/cm² and exposed to the directed

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beam at an equivalent fluence of 9.4×10^{20} atoms/cm². The plasma exposed sample appears to have a more porous epoxy surface remaining with the fibers loosely adherent to the underlying surface, while the epoxy surrounding the fibers in the directed beam exposed sample has taken on the highly filamentous and textured surface as observed with ram exposed polymers on Shuttle flight experiments in LEO [11]. The epoxy also seems to be more intact underneath the fibers due to the directionality of the oxidation.



a. Exposed in Air Plasma
Asher



b. Exposed in Atomic Oxygen
Directed Beam

Figure 5. Atomic Oxygen
Exposed Fiberglass-Epoxy

While the directed beam

exposure was performed on one side of the sample, the actual mast will experience a directed beam which will sweep all the way around it because the mast must rotate to maintain alignment of the solar cells with the sun. It would be expected that a more complete exposure of the surface would occur which would remove more of the epoxy underneath the glass fibers. The actual exposure is probably somewhere between the asher and the directed beam with the penetration depth of the atomic oxygen being much shallower for the beam and space exposures. Longer exposures with the beam are needed to verify this. The possibility of fiber removal would still be a serious problem.

The concern over the extent of fiber removal is severe enough that a means of coating or covering the surface may be needed to prevent the removal of fibers from the surface by either containing them at the very least, or preventing epoxy removal. The protection required must also be able to survive the surface strain levels (approximately 1%) experienced by the retracted longeron.

3.2 Durability Testing of Protected Fiberglass-Epoxy

All 5 types of protected longerons and an unprotected specimen were exposed to the air plasma for an equivalent fluence of approximately 4.6×10^{20} to 8.5×10^{20} atoms/cm² for initial durability evaluation. All coatings and coverings except the single aluminum braid appeared to provide some protection to the longeron (Figure 6).

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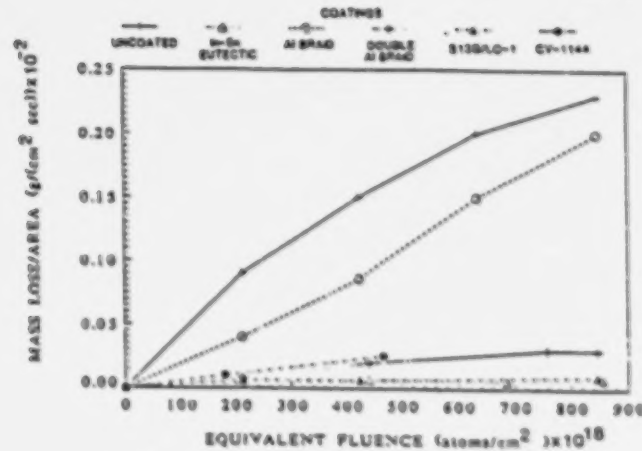


Figure 6. Mass Loss of Protected and Unprotected Fiberglass-Epoxy as a Function of Fluence

The single aluminum braid is very open which allows the fiberglass-epoxy to be directly exposed to the atomic oxygen. The braid openings are also large enough that the fibers could not be contained on the surface of the fiberglass-epoxy after they are exposed. The mass loss rates for the unprotected and aluminum braid protected fiberglass-epoxy composites are very similar and scanning electron micrographs of the surface under the braid were identical to those for the unprotected fiberglass-epoxy. The addition of a second braid layer reduced the mass loss rate further. The CV-1144 experienced some mass loss and this was attributed to areas where the paint did not completely cover the surface (Figure 7). This coating also seems to remain tacky after coating and exposure. The S13G/LO-1 and indium-tin eutectic coated samples experienced very little mass loss after exposure to atomic oxygen.

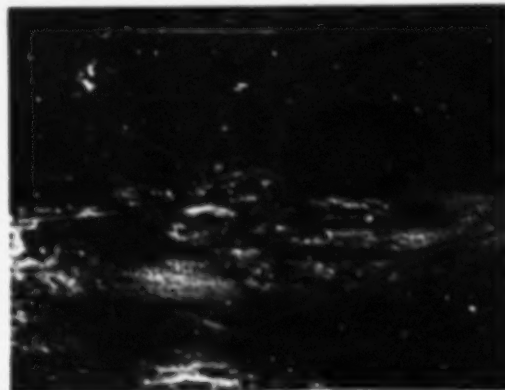


Figure 7. Scanning Electron Photomicrograph of a CV-1144 Coated Longeron After Exposure to an Atomic Oxygen Fluence of Approximately 4.6×10^{20} atoms/cm²

Longerons coated with CV-1144, In-Sn eutectic, and S13G/LO-1 were exposed to UV radiation followed by ashing in order to determine if UV could break down the protective nature of the coating. The 1000 ESH exposure appeared to have no effect on the mass loss rate of the longerons during ashing (Figure 8). A slight darkening of the S13G/LO-1 after UV exposure was the only observable

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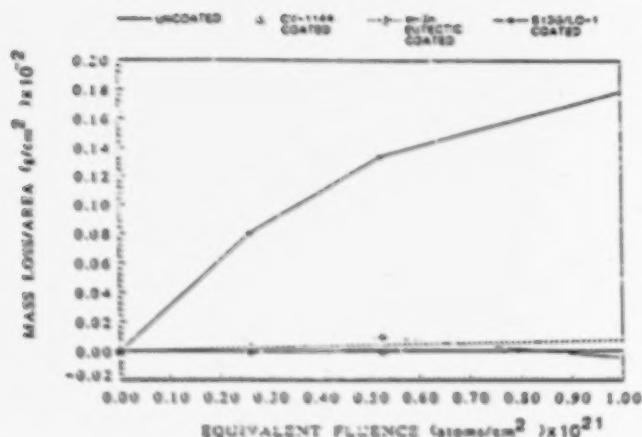


Figure 8. Mass Loss of Protected and Unprotected Fiberglass-Epoxy Exposed to an Air Plasma After 1000 ESH of VUV Exposure

effect. The CV-1144 performed slightly better in this test. This is possibly due to a more continuous coating on this sample.

Some of the protected samples and the unprotected sample from Figure 6 as well as a second unprotected sample were thermal cycled and then flexed before a second exposure to atomic oxygen in order to determine if the protective coatings could withstand these conditions and still protect against attack by atomic oxygen. Unprotected sample # 1 and the indium-tin eutectic coated sample were exposed at the same time. The remaining three were exposed together in a later exposure. Mass loss per unit area data for these exposures are shown in Figure 9.

The mass loss per unit area data for the unprotected and single aluminum braid protected longerons during the second atomic oxygen exposure agree fairly well with the data for the first atomic oxygen exposure.

This also appears to be true for the CV-1144 and the first 1.8×10^{20} atoms/cm² exposure of the indium-tin eutectic. The eutectic appears to lose a large amount of mass at greater fluence levels which is unexplained. The rate of loss is very close to that for unprotected epoxy, but there was no unprotected portion of the longeron visible as in the case of the CV-1144. There was also very little cracking of the surface observed. If the coating itself is being removed because it is being oxidized, the mass loss should have been observed earlier. S13G/LO-1 coated and double aluminum braid coated samples will be exposed in the future along with a verification exposure of the In-Sn eutectic.

4. CONCLUSIONS

Even though the total amount of epoxy removed from fiberglass-epoxy composites in low Earth orbit is not significant

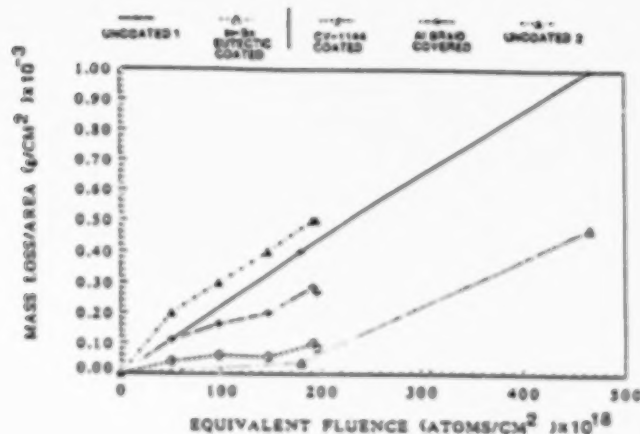


Figure 9. Mass Loss Per Unit Area for Unprotected and Protected Fiberglass-Epoxy Exposed to an Air Plasma After Exposure to Thermal Cycling and Flexure

for most applications, the contamination problems that could result from loss of glass fibers from the surface is of great concern. Preliminary tests with directed and random atomic oxygen sources indicate that fiber exposure is highly likely at the atomic oxygen fluence levels that Space Station will experience. Therefore some means of protection to either prevent loss of epoxy at best or provide fiber containment at the very least is needed. Of the coatings evaluated to date, CV-1144, S13G/LO-1, and In-Sn eutectic appear to provide good protection against atomic oxygen with a double aluminum braid covering being slightly worse. The single aluminum braid offered little to no protection and also seemed to provide no fiber containment ability. The ability to protect, however is very dependent on the continuity of the coating coverage. Vacuum

ultraviolet radiation appeared to have no effect on the ability of the coatings to resist atomic oxygen attack. Flexing and thermal cycling also appeared to have little effect on the mass loss rates of the braid covered samples and CV-1144 coating. The In-Sn eutectic coating exhibited an increase in mass loss which would indicate coating failure, however no visual indication of failure was observed. This coating and S13G/LO-1 will be tested in the future. CV-1144 appears to provide good fiber containment and atomic oxygen resistance if applied uniformly to the surface. S13G/LO-1 and In-Sn eutectic appear promising, however, more testing is needed.

5. ACKNOWLEDGEMENTS

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